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AN EVALUATION OF VAPOR-DEPOSITED TUNGSTEN TUBING

by

R. G. Mills, J. R. Lindgren, and A. F. Weinberg

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TOPICAL REPORT

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Sponsored by

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INTRODUCTION

An evaluation of commercially available vapor-deposited tungsten tubing was initiated during April, 1964. This is part of the continuing study that General Atomic is making on the development of vapor-deposited tungsten. The tests performed on 17 tubes included analyses for impurity content, resistance to grain growth during various heat treatments, hardness measurements, and ductile-to-brittle transition temperature. A significant portion of this report is a series of photomicrographs of the tubing before and after four heat treatments (Figs. 1 through 17).*

MATERIALS RECEIVED

An invitation was sent to fourteen organizations to supply tubing for evaluation, and eight responded by sending material. A copy of the letter of invitation is given in Appendix A.

Seventeen tubes were received from the eight organizations. They ranged from 1/4 to 1/2 in. in diameter, from 2 to 8 in. in length, and from 0.010 to 0.045 in. in wall thickness. The dimensions and weight of each tube are listed in Table 1.

The methods of forming varied greatly (see Table 2). Nine tubes were vapor-deposited, seven were extruded, and one was electrodeposited. One vapor-deposited tube was deposited by hydrogen reduction of the chloride, one was deposited from WF_6 that had been converted from WCl_6 (Tube 17), and the remainder of the tubes were deposited by hydrogen reduction of the fluoride; the electrodeposited tube was deposited from a fused salt bath.

EVALUATION PROCEDURE

All tubes were weighed, measured, and photographed as-received. Samples of each tube submitted were analyzed for impurity content, and an as-received microstructure and hardness determination was made. Additional pieces were cut and each was subjected to one of four heat treatments: 1800°C for 100 hr, 2000°C for 15 hr, 2200°C for 15 hr, and 2500°C for 1 hr. This procedure was carried out on the first sixteen tubes at one time; Tube 17 was evaluated later. A number of rings (3/16 in. wide) were cut from each tube and compressed at various temperatures in an Instron

*All figures are placed at the end of the report.

Table 1

AS-RECEIVED DIMENSIONS OF TUNGSTEN TUBES

GA Tube No.	Mfgr. Code	Dimensions (in.)			Weight (g)
		Length	OD	Wall Thickness	
1	A	5.65	0.377	0.015	26
2	A	5.85	0.377	0.015	29
3	B	2.02	0.400	0.007-0.015	9
4	C	5.35	0.271	0.030-0.045	46
5	D	5.95	0.378	0.015	30
6	D	6.00	0.378	0.015	32
7	E	6.15	0.420	0.025	51
8	E	7.80	0.402	0.016	51
9	F	3.52	0.470	0.040	50
10	F	3.47	0.450	0.040	42
11	F	3.56	0.500	0.045	71
12	F	3.52	0.495	0.045	68
13	F	6.00	0.375	0.045	89
14	F	5.97	0.375	0.045	88
15	F	5.92	0.375	0.045	89
16	G	6.03	0.375	0.015	32
17	H	4.00	0.350	0.014-0.022	30

NOTE: Dimensions and weights are those determined by General Atomic.

testing machine to determine the ductile-to-brittle transition temperature. The outside of all tubes was electropolished in a sodium-hydroxide bath to achieve a smooth finish before compression testing. The compression testing was done in an air atmosphere.

Table 2

FORMING-METHOD VARIABLES

Tube No.	Mfgr. Code	Forming Method	Mandrel		Layers	
			Male	Female	Uninterrupted	Interrupted
1	A	Vapor-deposited		x	x	
2	A	Vapor-deposited		x	x	
3	B	Electrodeposited	x		x	
4	C	Vapor-deposited		x		x
5	D	Vapor-deposited		x		x
6	D	Vapor-deposited		x	x	
7	E	Vapor-deposited	x		x	
8	E	Vapor-deposited		x	x	
9	F	Extruded at 1800°C				
10	F	Extruded at 1800°C				
11	F	Extruded at 1200°C				
12	F	Extruded at 1200°C				
13[a]	F	Extruded, 6 to 12 in., at 1200°C				
14[a]	F	Extruded, 12 to 18 in., at 1200°C				
15[a]	F	Extruded, 18 to 24 in., at 1200°C				
16	G	Vapor-deposited	x			x
17	H	Vapor-deposited	x		x	

[a] Tubes 13, 14, and 15 are three sections of one tube extruded at 1200°C; Tube 13 is the 6 to 12 in. section from the lead end, and 14 and 15 are subsequent 6 in. sections.

The only exceptions to the above procedure were Tube No. 3, which had to be compression-tested before the impurity analysis was run, and Tube No. 17, which was received too late to determine the ductile-to-brittle transition temperature.

RESULTS

The gas impurity content, carbon content, and fluorine content are listed in Table 3. The metallic impurity content determined by emission spectrography is listed in Table 4. Appendix B is a brief description of the analytical methods used in these determinations.

Table 3

FLUORINE, CARBON, NITROGEN, OXYGEN, AND HYDROGEN CONTENT OF TUNGSTEN TUBING
(In ppm)

Tube No.	Mfgr. Code	F_2	C	N_2	O_2	H_2
1	A	29.3	7.7-6.7	10, 7	10, 16	3, 7
2	A	46	12.5-10.0	9, 10	8, 7	3, 3
3	B	13	-----	8, 7	105, 116[a]	6, 6
4	C	26	6.7	2, 5	3, 6	2, 3
5	D	44.4	6.1-7.3	3, 6	32, 37	4, 4
6	D	35	6.6-6.5	1, 2	7, 10	4, 4
7	E	9	6.4-6.0	5, 5	6, 7	3, 3
8	E	4	6.2-6.5	5, 3	12, 11	4, 4
9	F	3	11.0-11.6	11, 10	22, 20	4, 4
10	F	4	7.9	20, 14	6, 7	2, 2
11	F	4	6.7-7.6	13, 14	6, 19	2, 2
12[b]	F					
13	F (6 to 12 in.)	3	5.7-6.5	20, 15	8, 9	3, 3
14[b]	F (12 to 18 in.)					
15[b]	F (18 to 24 in.)					
16	G	22 (Cl_2)	6.1	5, 8	12, 20	2, 3
17	H	18	23, 22	<1, 1	4, 8	10, 5

[a] There was an oxide film on the inside of the tube.
[b] Samples 12, 14, and 15 were not submitted for analysis.

The microstructures of all tubes before and after heat treatments are illustrated in Figs. 1 through 17. The hardness measurements made on these samples are listed in Table 5 and shown in Fig. 18. The ductile-to-brittle transition temperatures are listed in Table 6.

The data are interpreted under "Discussion of Results," below.

Table 4

METALLIC IMPURITY CONTENT OF TUNGSTEN TUBING
(In ppm)

Tube No.	Mfgr. Code	Al	Cu	Fe	Mg	Mn	Mo	Ni	Si	Ti
1	A	<1	N<0.5[a]	<1	0.5	N<0.5	N<100	1	<2	N<6
2	A	N<1	<0.5	<1	0.5	N<0.5	N<100	<1	<2	N<6
3	B	4	600	2	<0.5	N<0.5	800	200	2	N<6
4	C	<1	1	20	1	1	N<100	4	6	N<6
5	D	<1	<0.5	1	<0.5	N<0.5	N<100	<1	<2	N<6
6	D	6	<0.5	100	0.5	N<0.5	N<100	4	2	N<6
7	E	2	N<0.5	<1	0.5	N<0.5	N<100	N<1	<2	N<6
8	E	1	N<0.5	<1	0.5	N<0.5	N<100	<1	2	N<6
9	F	4	1	20	0.5	0.5	N<100	2	8	N<6
10	F	3	N<0.5	80	<0.5	N<0.5	N<100	200	<2	N<6
11	F	2	<0.5	60	<0.5	1	100	10	<2	N<6
12[b]	F									
13	F (6 to 12 in.)	1	<0.5	10	0.5	N<0.5	200	8	2	N<6
14[b]	F (12 to 18 in.)									
15[b]	F (18 to 24 in.)									
16	G	2	<0.5	10	1	N<0.5	N<100	<1	10	N<6
17	H	2	<0.5	5	<0.5	1	N<100	200	8	<6

NOTE: Elements not listed above were not detected in any of the samples.

[a] N = Not detected.

[b] Samples 12, 14, and 15 were not submitted for analysis.

Table 5

MICROHARDNESS OF AS-RECEIVED TUNGSTEN TUBING

Tube No.	Mfgr. Code	Hardness (15 g)			Hardness (100 g)		
		High	Low	Average	High	Low	Average
1	A	385	359	374	370	363	366
2	A	394	368	384	401	376	389
3	B	376	351	367	397	348	368
4	C	344	290	310	425	401	416
5	D	351	302	316	473	441	454
6	D	394	329	364	420	394	409
7	E	308	262	283	397	357	373
8	E	368	359	363	409	397	405
9	F	308	296	303	373	336	360
10	F	315	302	306	405	394	398
11	F	404	385	394	454	441	446
12	F	344	315	328	401	383	389
13	F						
	6 in.	376	278	316	401	383	395
	12 in.	344	290	320	483	450	466
14	F (18 in.)	344	290	324	429	401	414
15	F (24 in.)	308	257	277	519	488	502
16	G	315	284	299	373	348	358
17	H	359	290	335	363	306	342

NOTE: The values listed are Diamond Pyramid Hardness determined under both a 15-g load and a 100-g load on transverse sections; five determinations were made at each load.

DISCUSSION OF RESULTS

Chemical Impurity Content

The fluorine content of extruded tubing was in the range of 3 to 4 ppm and was probably in the tungsten powder used to fabricate the extrusion blank. The fluorine content of tubing made by the vapor-deposition of WF_6 was in the range 15 to 40 ppm, with the exception of tubing from manufacturer E, which contained 4 and 9 ppm. These latter values are just slightly higher than those for the extruded tubing and indicate little or no pickup of fluorine during the vapor-deposition process.

Most of the carbon contents were 4 to 7 ppm with the following exceptions: Tube 17 contained 22 ppm, Tube 9 contained 11 ppm, and

Tube 2 contained 12 ppm. Values for oxygen content ranged from 3 to 22 ppm for all tubes, except Tube 3, which contained 110 ppm, and Tube 5, which contained 35 ppm. The probable cause for the high value in Tube 3 was the thin film of brown oxide on the inside of the tube, which was noticed during the transition-temperature heating of the specimen. A second specimen, from which this film was cleaned, was analyzed for metallic impurities (the silicon content dropped from 20 to 2 ppm), but sufficient sample was not available for reanalysis of the oxygen content.

The probable reason for the high oxygen content of Tube 5 was that deposition was purposely interrupted six times, and each boundary can readily become the site for gaseous impurities (see Fig. 5).

Values for hydrogen parallel those for oxygen; most were 2 to 4 ppm, with two exceptions: Tube 3 contained 6 ppm, and Tube 17 contained 5 and 10 ppm. Values for nitrogen were determined by the difference method, and values ran from 1 to 20 ppm, with no evident correlation to other gases.

Thirty-three metallic elements were sought by analysis with the emission spectrograph. Nine metallic elements were found and are listed in Table 4. Values for these elements which are significantly higher than the detectable limits are probably related to the mandrel material used during the deposition. (Work performed at General Atomic has indicated that if the surface of the deposit in contact with the substrate is removed by mechanical or chemical techniques, these concentrations of impurities can often be lowered.) Tube 17 was made on a nickel mandrel, and the impurity analysis indicated 200 ppm nickel. No other manufacturer indicated the mandrel material used.

Hardness Measurements

Microhardness measurements of as-received tubing varied with the test load used. Tube 15, for example, had the highest hardness value measured with the 100-g load and the lowest hardness value measured with the 15-g load (see Fig. 18 and Table 5). In all cases the hardness value determined with the 100-g load was the same or higher than the value determined with the 15-g load, but there was a wide spread in many cases.

Since light loads did not penetrate as deeply into the specimens as the heavier loads, the boundaries of the grain that was subjected to the light load did not influence the penetration. When heavier loads were used, the measured hardness was structure-sensitive because the depth of penetration was affected by the grain boundaries.

Ductile-to-brittle Transition Temperature

The ductile-to-brittle transition temperature in this work is defined as the temperature at which the first easily measurable deformation was noticed. A bend in the stress-strain curve after initial loading indicated that deformation was occurring.

Some of the rings that were compressed broke at the 6 and 12 o'clock positions prior to any deformation, but the resulting half rings deformed under less load with no increase in test temperature. Failure of rings under the platen was noted only for tubing made on a female mandrel or the thick-walled extruded tubing. This phenomenon was no doubt related to stress concentrations existing on the interior diameter of the tube, which was not electropolished. It is felt that the temperature at which deformation can be obtained prior to failure at the 3 and 9 o'clock positions is more meaningful data for determination of the ductile-to-brittle transition and it is this value that is reported in Table 6.

Table 6

DUCTILE-TO-BRITTLE TRANSITION TEMPERATURE

Tube No.	Mfgr. Code	Lowest temp. at which any Deformation Occurred (^{o}C)
1	A	260-325
2	A	365-375
3	B	<180
4	C	285[a]
5	D	240-270[a]
6	D	170
7	E	175-200
8	E	140[a]
9[b]	F	
10	F	130[a]
11[b]	F	130[a]
12[b]	F	
13	F (6 to 12 in.)	>160[a]
14[b]	F (12 to 18 in.)	
15[b]	F (18 to 24 in.)	
16	G	260-270

[a]These values were determined on split rings that fractured in a brittle fashion at the 12 and 6 o'clock positions. Higher temperatures were required to obtain deformation without fracture of the full ring.

[b]Samples 9, 12, 14, and 15 were not submitted for this evaluation.

A correlation was made of the transition temperature and the fluorine content. This is indicated in Fig. 19. The curve shows little scatter except for Tubes 5 and 6. The fluorine may be present within these specimens in a different form, as evidenced, perhaps, by the significant amount of porosity found in these specimens after heat treatments at 2200^{o} and 2500^{o}C. However,

even in these specimens the higher of the two fluorine contents were associated with the higher ductile-to-brittle transition temperature.

The ductile-to-brittle transition temperature as a function of heat treatment will be an area of interst for further study.

The breaking strengths required to cause failure in the ring compression tests were in the range of 100,000 to 250,000 psi for most vapor-deposited tubing, and 250,000 psi for extruded tubing. Tubes 1, 2, and 16 were noticeably weaker than the others, requiring 100,000 to 150,000 psi. This was also noticed qualitatively in handling these tubes.

Microstructure

Vapor-deposited tungsten generally resists grain growth to a greater degree than does wrought material. When grain growth does occur in vapor-deposited tungsten during heat treatment, it originates where there are small randomly oriented grains, such as at the surface of initial deposition, rather than in the highly oriented columnar grain structure. In this report, the criterion for evaluating a structure is its resistance to grain growth. The degree of grain growth, the degree of retention of a columnar structure, and whether or not the grain boundaries extend entirely through the wall thickness were evaluated.

All photomicrographs taken of the 2000°C heat treatment show a "film" on one or both surfaces of the piece. This film is probably a second phase formed by an impurity in the furnace atmosphere during the heat treatment; however, the grain structure appears to be unaffected by the presence of this impurity layer. Tube 17, which was heated separately, does not show this film.

Many structures show smaller grains after the 2500°C, 1-hr heat treatment than after the 2200°C, 15-hr heat treatment. Probably because of the shorter time, the higher-temperature heat treatment has not produced as much energy input and the grains are still in the process of growing. The microstructures are discussed in more detail below.

Tubes 1 and 2. These tubes from manufacturer A appeared to be similar to each other, and they retained a partially columnar structure after all heat treatments. Grain growth was initiated on the initial deposition surface but did not penetrate more than one-half of the wall thickness. Porosity was evident after the 2200°C heat treatment.

Tube 3. This tube was electroformed and had a randomly oriented structure in the as-received condition. The grain growth was extensive after the first heat treatment at 1800°C, and by 2000°C individual grains extended through the wall thickness.

Tube 4. There was an interruption boundary in this tube, and considerable grain growth occurred within the initial layer. The material deposited after this layer was formed remained columnar after all heat treatments. This boundary was not evident in the as-received condition in a thicker section taken from the same tube.

Tubes 5 and 6. Each of these tubes from manufacturer D was fabricated in a different way. Tube 5 was seven-layered, with the interrupted layers very well defined in the as-received condition. The structure of the initial layer was carried in a radial direction to the inside diameter. There was evidence of gross porosity at the boundaries of interruption after the heat treatments. Grain growth occurred within a deposition layer, and the columnar structure was destroyed. Tube 6 was a single layer and the columnar structure remained after all heat treatments, although lateral growth took place at the substrate interface. Some porosity was seen after the 2500°C heat treatment.

Tube 7. This tube from manufacturer E had the most stable structure of the 17 tubes evaluated. There was very little grain growth after any heat treatment. The fine grains which were initially deposited were absorbed into the columnar structure.

Tube 8. This tube, also from manufacturer E, was deposited inside a female mandrel. Extensive grain growth took place during heat treatment, and at 2200°C the grains extended completely through the wall thickness. The impurity contents of Tubes 7 and 8 were very similar, but the structures as-deposited and after heat treatment were vastly different. Tube 7 had a highly oriented columnar structure as-deposited, which was retained after all heat treatments; Tube 8 had a more randomly oriented structure as-deposited and suffered extensive grain growth during all heat treatments.

Tubes 9, 10, 11, and 12. These tubes were extruded tubes. Tubes 9 and 10 were extruded at 1800°C and Tubes 11 and 12 at 1200°C. Extensive grain growth of Tube 10 occurred at 1800°C, whereas Tube 9 did not experience massive grain growth until 2200°C. Tubes 11 and 12 had very fine-grain structures in the as-received condition, but the grains penetrated completely through the walls during the 1800°C heat treatment.

Tubes 13, 14, and 15. These tubes were three 6-in. pieces cut from one 18-in. tube extruded at 1200°C. The only difference observed in the structure was that Tube 15 (last portion to be extruded) showed massive grain growth at a lower heat-treatment temperature (1800°C) than Tubes 13 and 14 (2000° to 2200°C).

Tube 16. This tube was the only one that was formed by WCl_6 deposition. There was no evidence in the as-received structure of the

interruption layer which was evident after each heat treatment. Some evidence of columnar structure remained even after 2500°C, although massive grains did extend through the wall thickness.

Tube 17. This tube showed a randomly oriented structure in the as-received condition, which became more regular after the 1800°C heat treatment. Massive grain growth appeared after 2200°C, with the grains extending through the wall thickness.

CONCLUSIONS

1. The chemical impurity content of vapor-deposited tubing from six sources is comparable. This is surprising considering that different starting materials, different experimental conditions, and different personnel were involved.

2. The metallic impurities are at a low level in all samples. The impurities present are related to the mandrel material.

3. There is a positive correlation between the residual fluorine content and the ductile-to-brittle transition temperature of vapor-deposited tungsten tubing. This merits further investigation, such as the evaluation of tubing made by the same technique but with various fluorine contents (see Fig. 19).

4. Extruded tungsten tubing has a very low fluorine content, but fluorine is present. The source of this fluorine is probably the tungsten starting material.

5. The lowest fluorine content of the vapor-deposited tubing (Tube 8) is comparable to that found in the extruded tubing made by powder metallurgy techniques.

6. The grain growth of Tube 7, formed on the male mandrel, exhibited the least change after heat treatments.

7. Columnar structures which are deposited in a highly oriented manner will exhibit less grain growth than those deposited in a less oriented manner. When grain growth does occur, it is initiated in the fine grains first deposited.

8. Grain growth of vapor-deposited tubing is not primarily related to the chemical impurity content. Tubes 7 and 8 were comparable in impurity content but had vastly different structures after heat treatment.

9. Extensive grain growth took place in the extruded tubing during heat treatment.

10. The ductile-to-brittle transition temperatures of extruded tungsten tubing and of the best vapor-deposited tubing are comparable.

11. No correlation was found between hardness and any other variable, i. e., impurity content, microstructure, or ductile-to-brittle transition temperature.

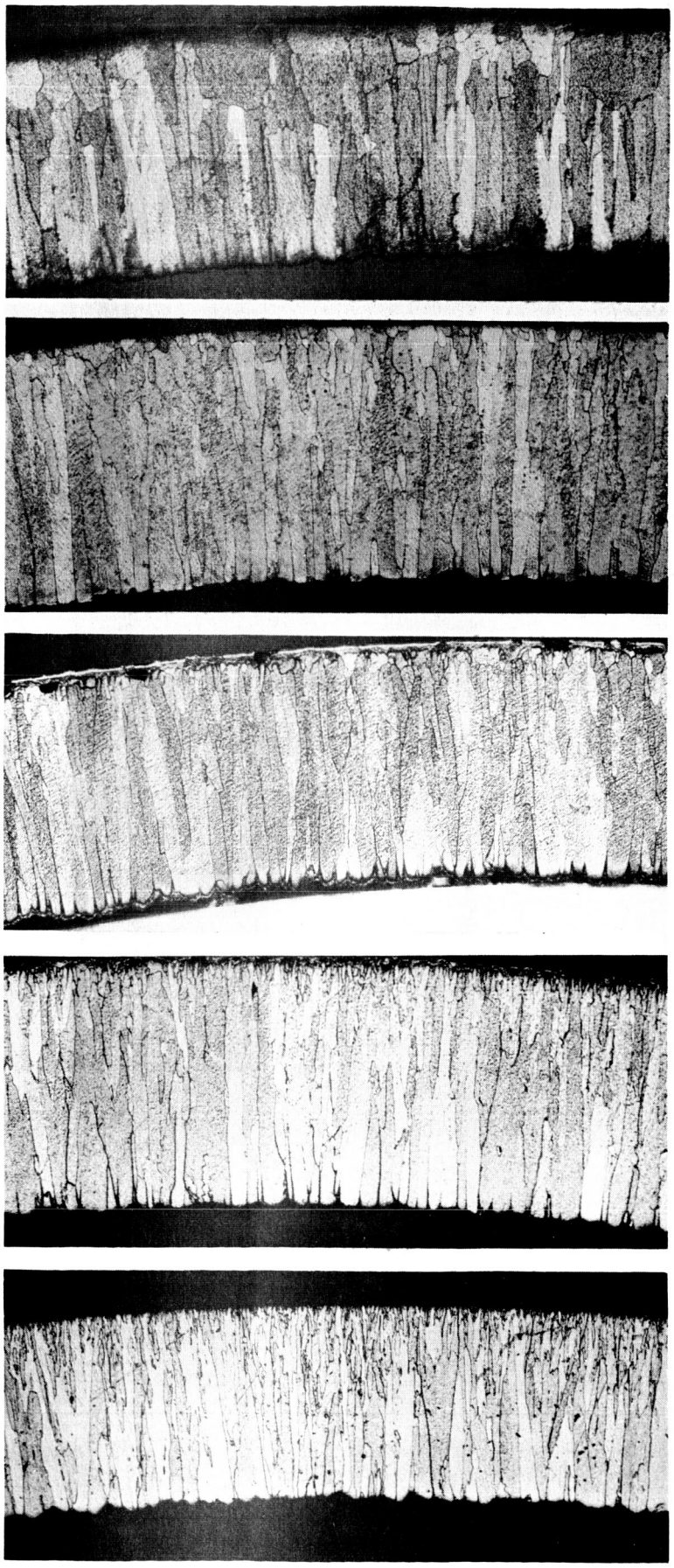
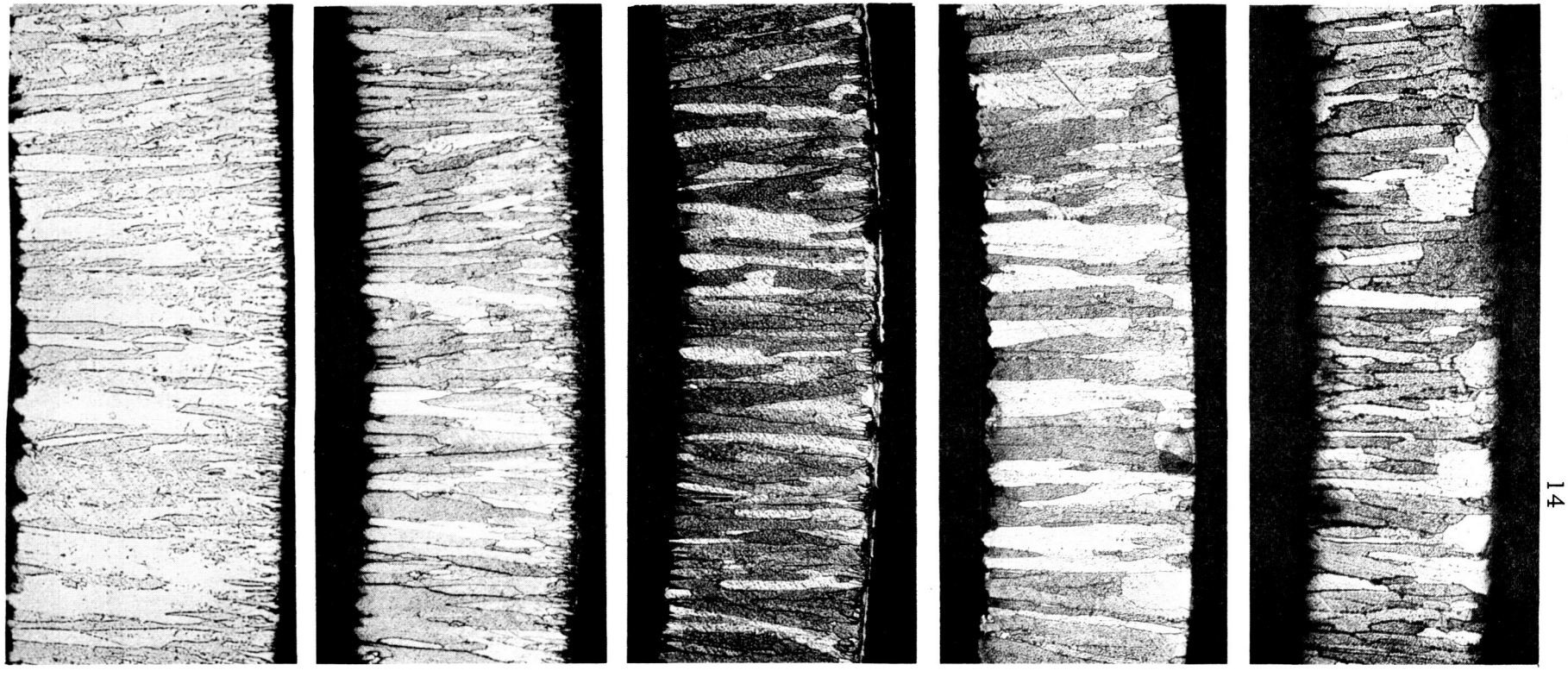


Fig. 1--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 1



M-7775-2 (100×) As-received

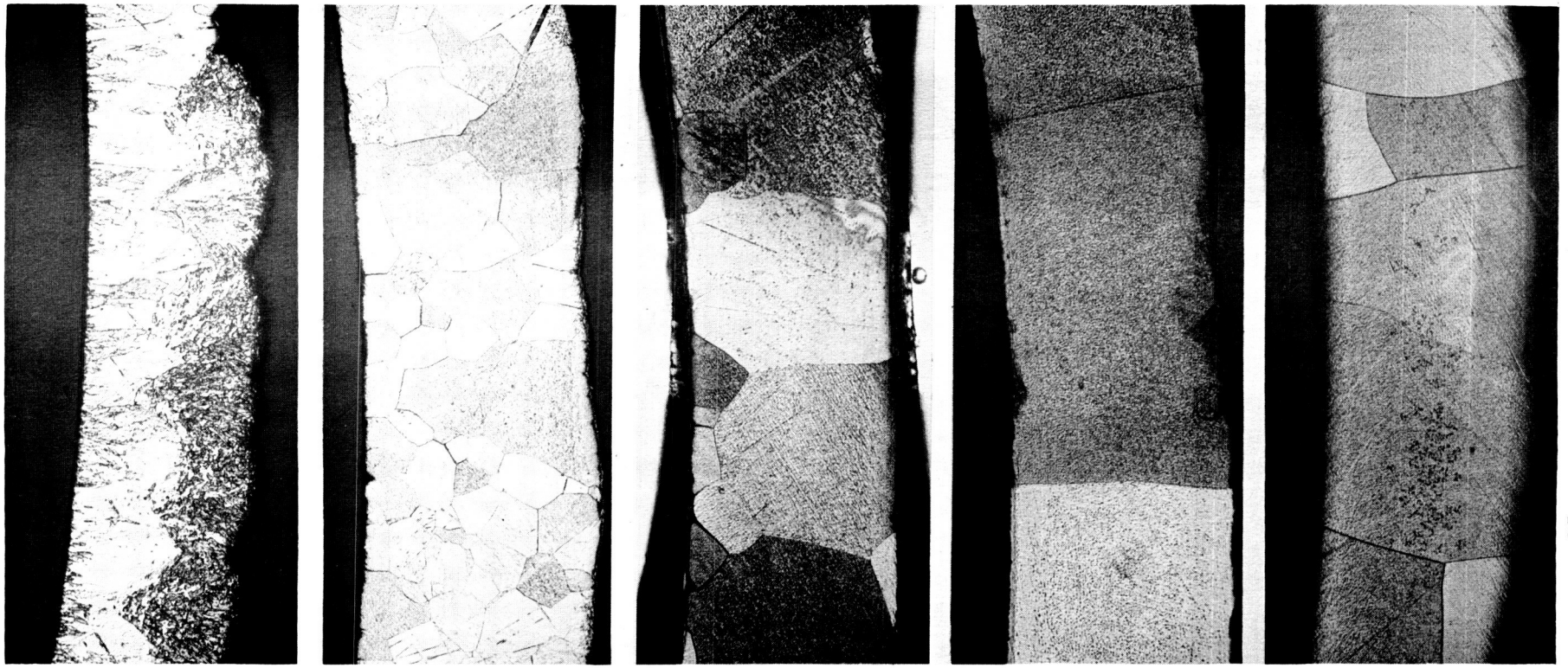
M-8244-1 (100×) 1800°C-100 hr

M-8474-1 (100×) 2000°C-15 hr

M-8532-1 (100×) 2200°C-15 hr

M-8579-1 (100×) 2500°C-1 hr

Fig. 2--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 2



M-7776-2 (100×) As-received

M-8245-1 (100×) 1800°C-100 hr

M-8475-1 (100×) 2000°C-15 hr

M-8533-1 (100×) 2200°C-15 hr

M-8580-1 (100×) 2500°C-1 hr

Fig. 3--Effect of thermal treatment on electrodeposited tungsten--Tube No. 3

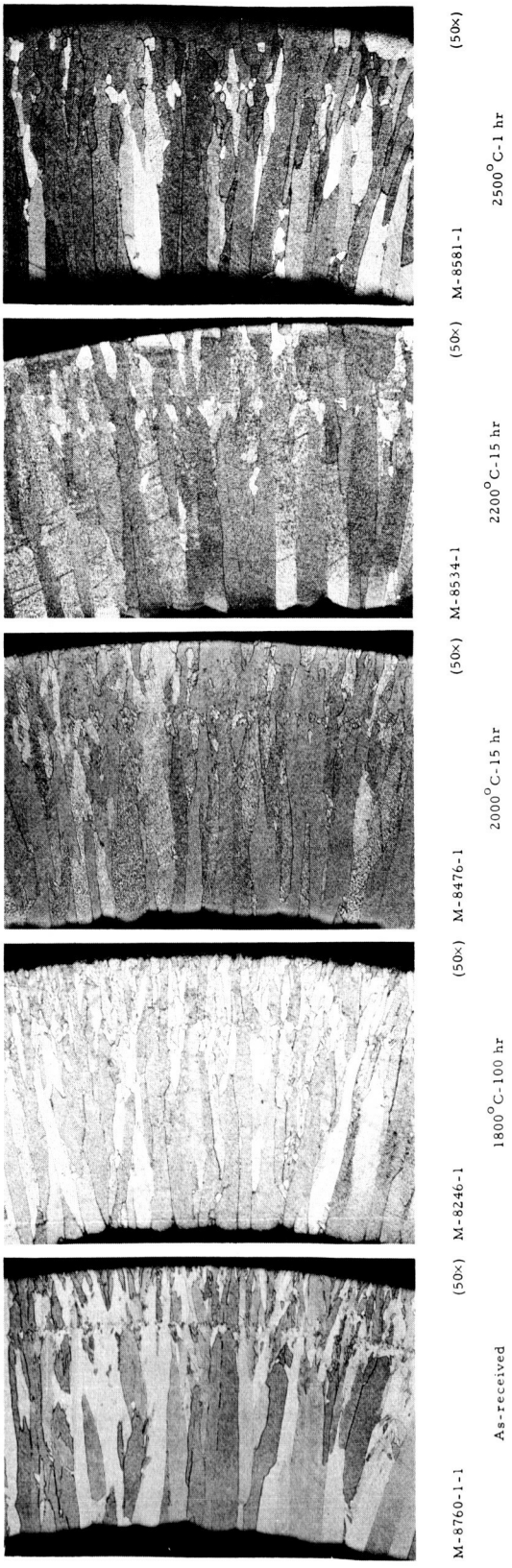
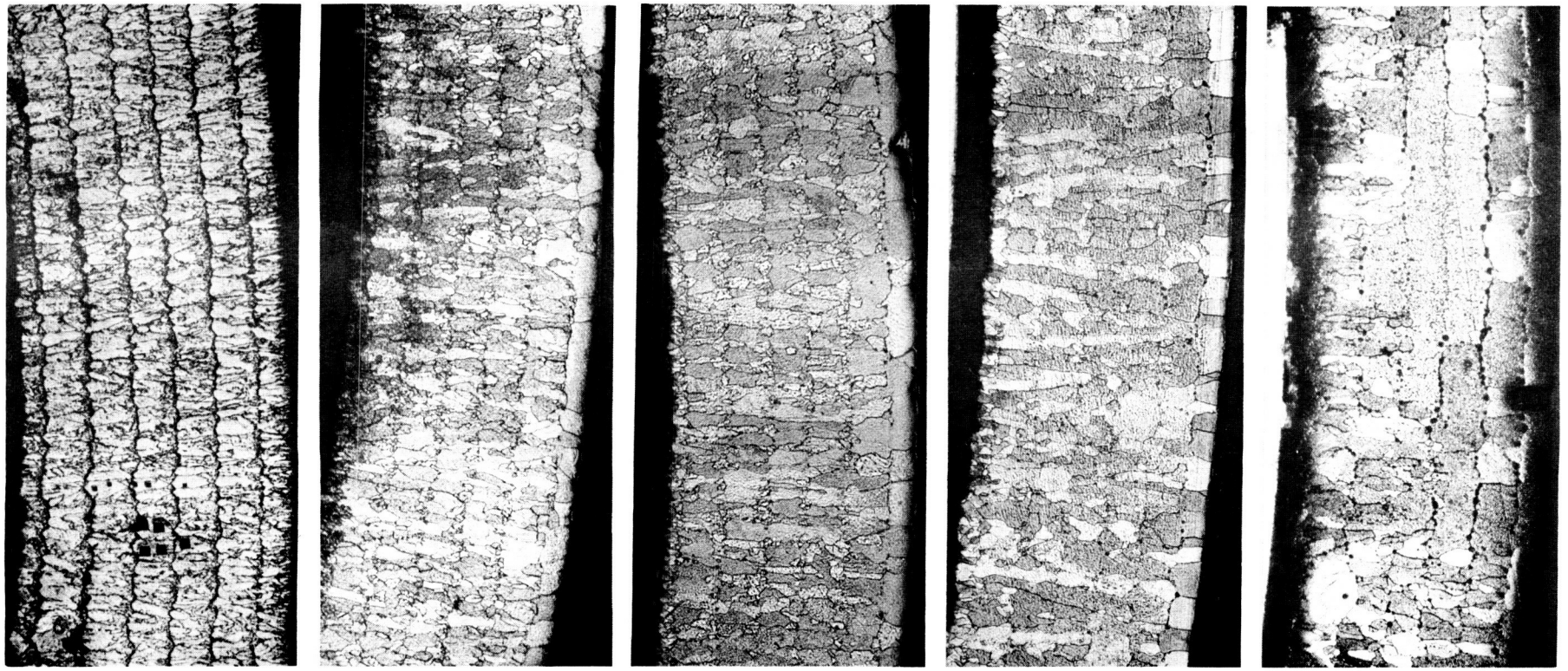


Fig. 4--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 4



M-7955-1 (100×) As-received

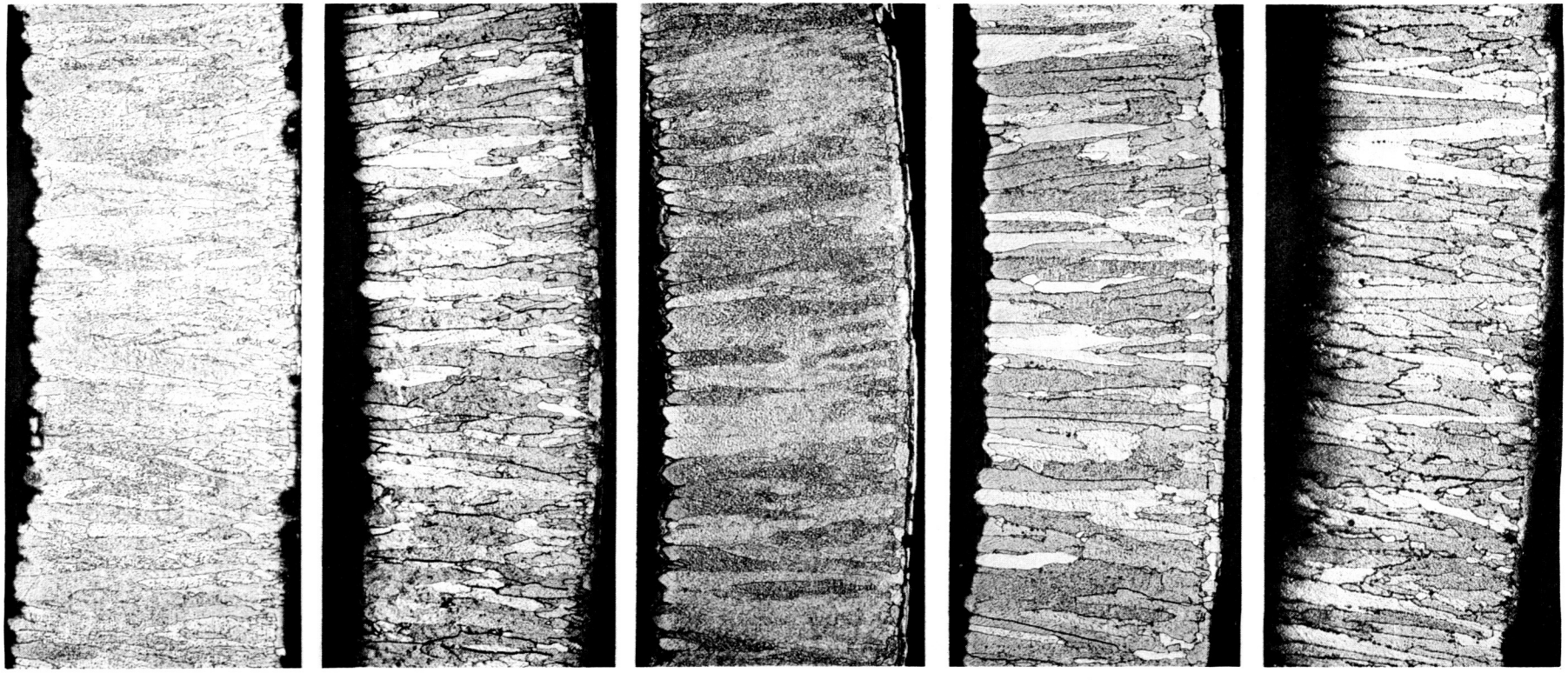
M-8247-1 (100×) 1800°C-100 hr

M-8477-1 (100×) 2000°C-15 hr

M 8535-1 (100×) 2200°C-15 hr

M-8582 (100×) 2500°C-1 hr

Fig. 5--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 5



M-7956 (100×) As-received

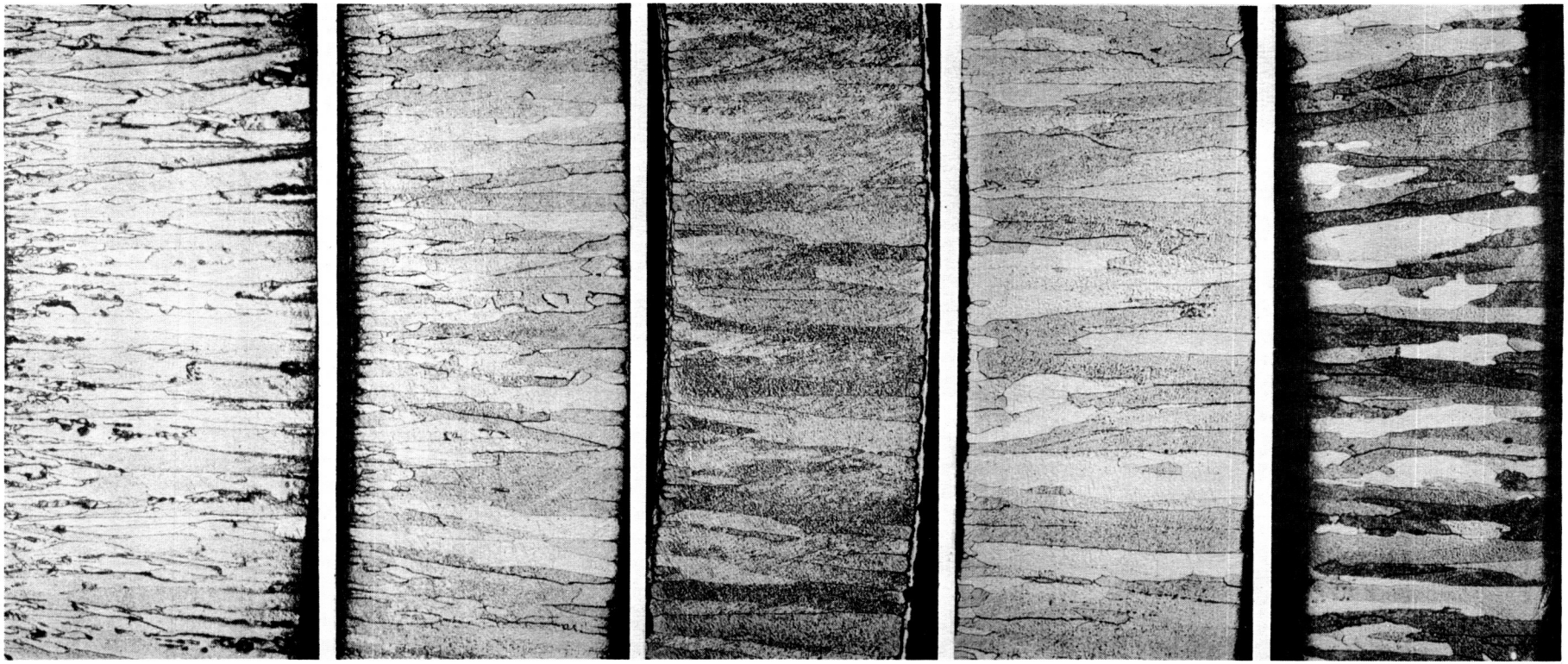
M-8248-1 (100×) 1800°C-100 hr

M-8478-1 (100×) 2000°C-15 hr

M-8536-1 (100×) 2200°C-15 hr

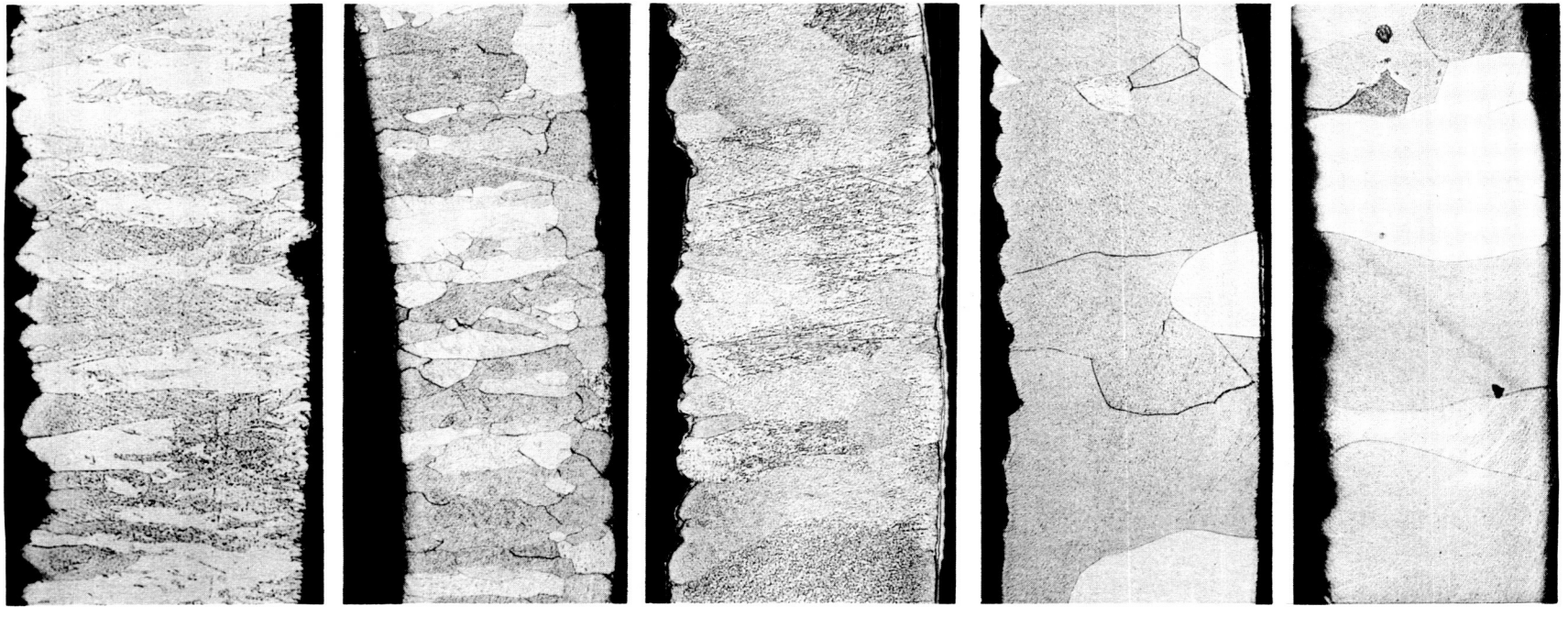
M-8583-1 (100×) 2500°C-1 hr

Fig. 6--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 6



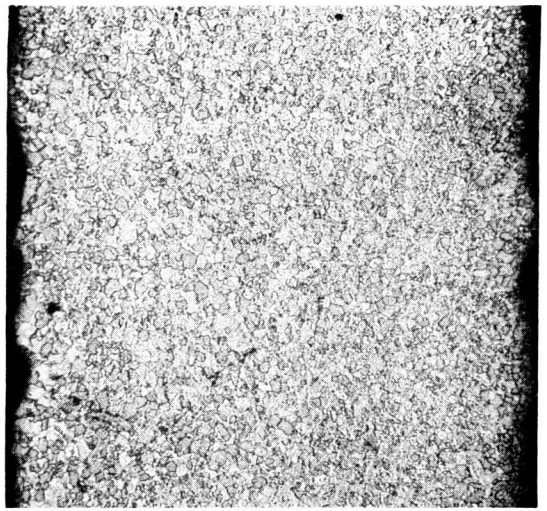
M-8176-3 (100×) As-received | M-8249-1 (100×) 1800°C-100 hr | M-8479-1 (100×) 2000°C-15 hr | M-8537-1 (100×) 2200°C-15 hr | M-8584-1 (100×) 2500°C-1 hr

Fig. 7--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 7



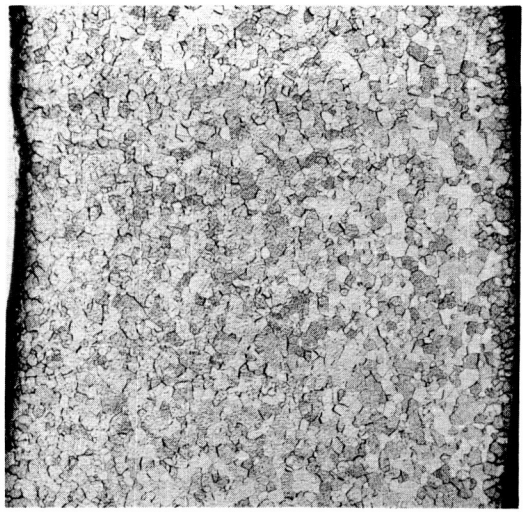
M-7778-2 (85×) As-received | M-8250-1 (85×) 1800°C-100 hr | M-8480-1 (85×) 2000°C-15 hr | M-8538-1 (85×) 2200°C-15 hr | M-8585-1 (85×) 2500°C-1 hr

Fig. 8--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 8



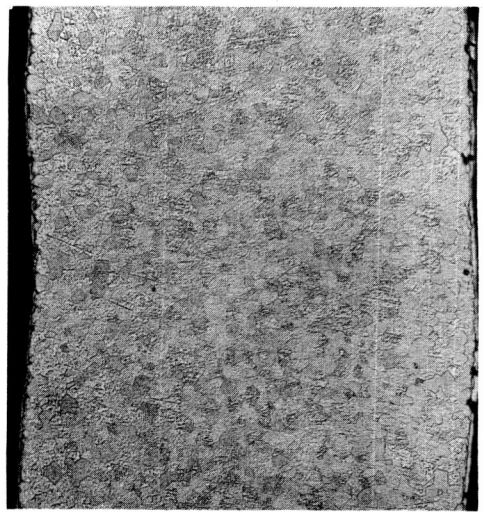
M-3813-2 (75×)

As-received



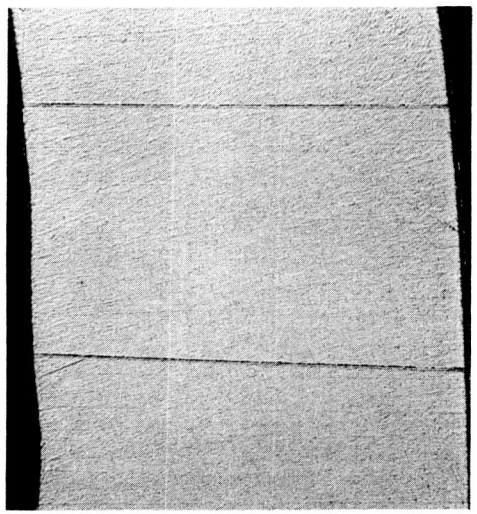
M-8251-1 (75×)

1800°C-100 hr



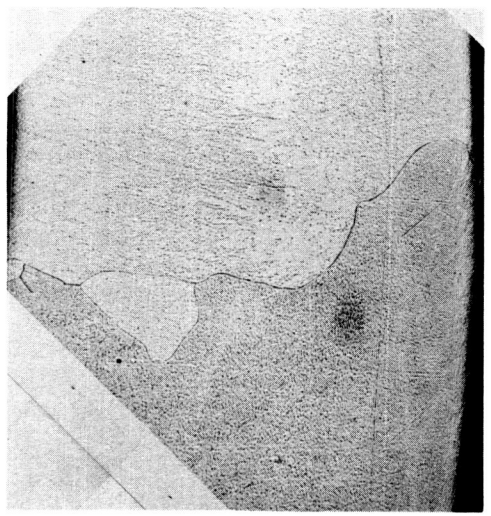
8481-1 (75×)

2000°C-15 hr



M-8539-1 (75×)

2200°C-15 hr



M-8586-1 (75×)

2500°C-1 hr

Fig. 9--Effect of thermal treatment on tungsten extruded at 1800°C--Tube No. 9

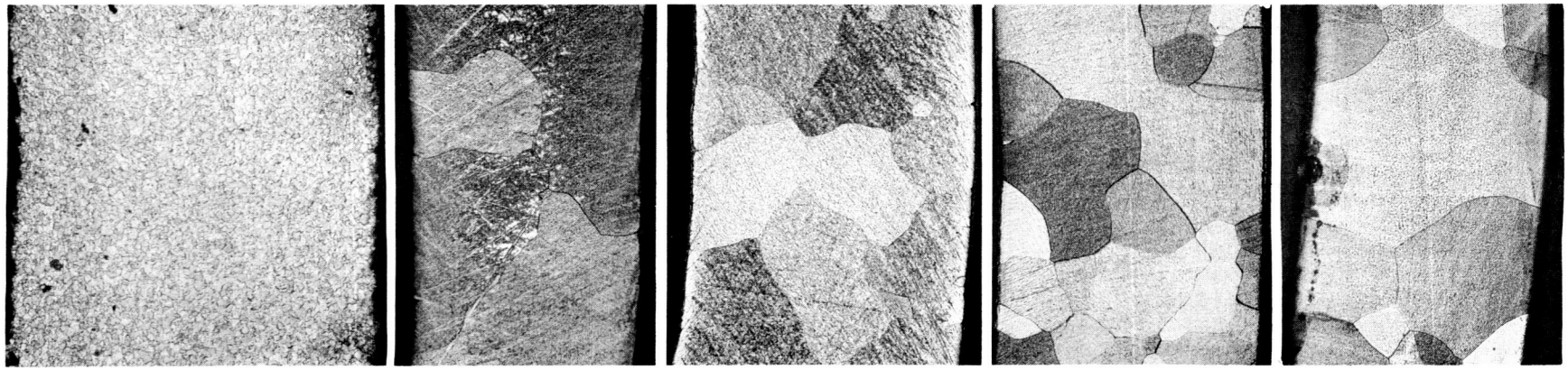


Fig. 10--Effect of thermal treatment on tungsten extruded at 1800°C--Tube No. 10

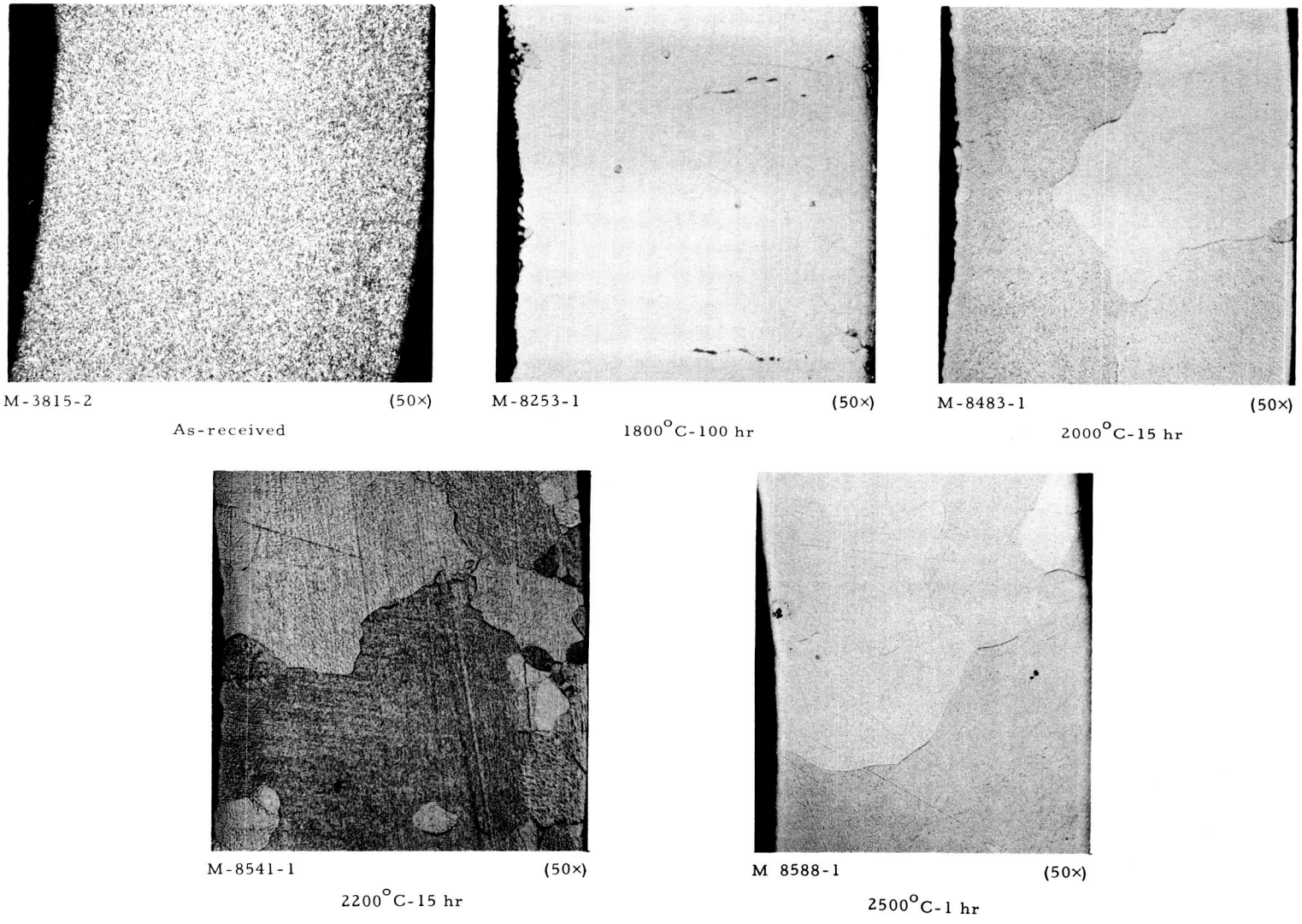
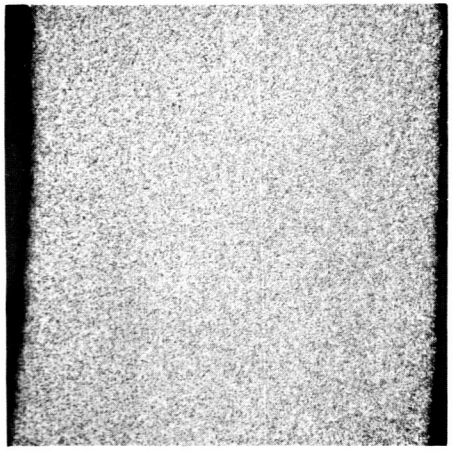
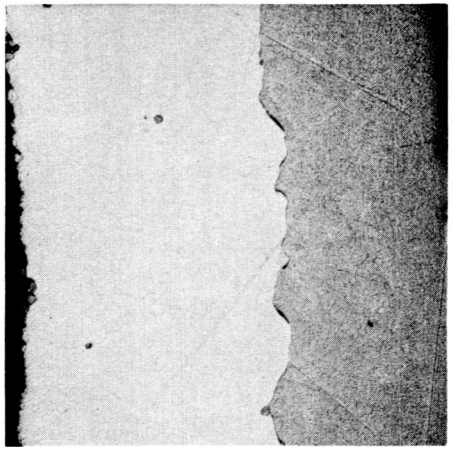


Fig. 11--Effect of thermal treatment on tungsten extruded at 1200°C--Tube No. 11



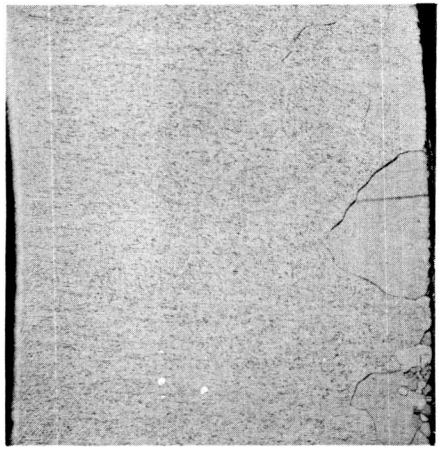
M-3816-2 (50×)

As-received



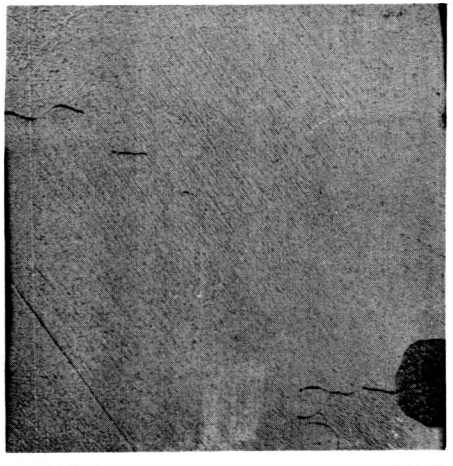
M-8254-1 (50×)

1800°C-100 hr



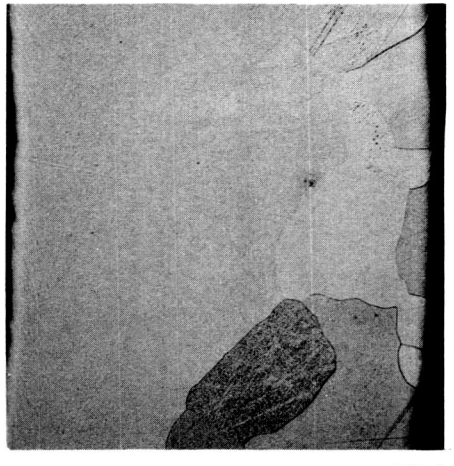
M-8484-1 (50×)

2000°C-15 hr



M-8542-1 (50×)

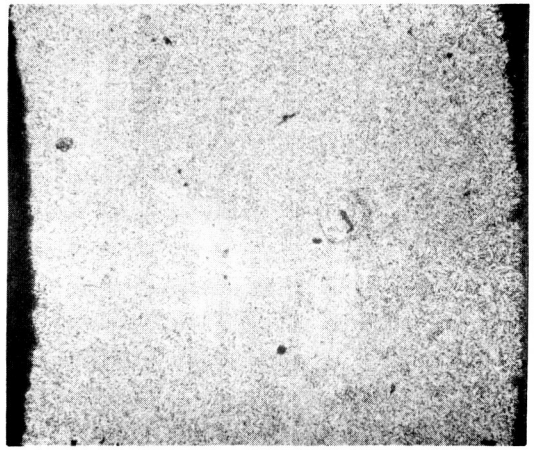
2200°C-15 hr



M-8589-1 (50×)

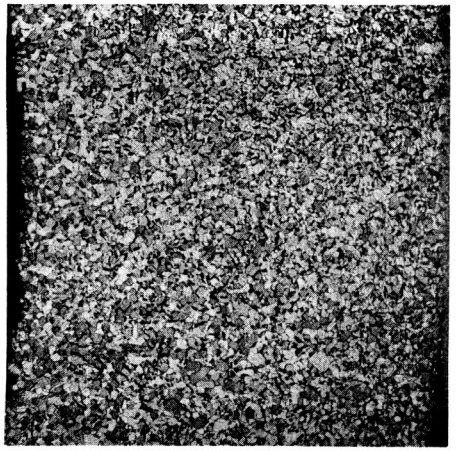
2500°C-1 hr

Fig. 12--Effect of thermal treatment on tungsten extruded at 1200°C--Tube No. 12



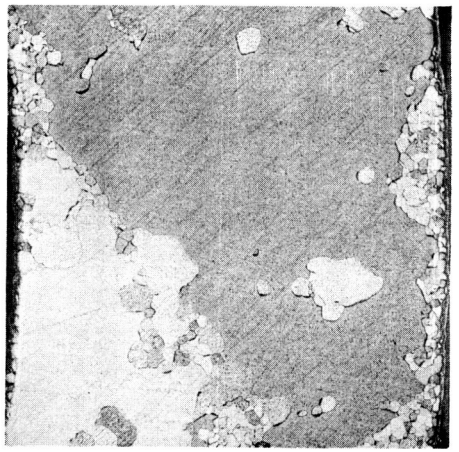
M-7962-1 (50×)

As-received



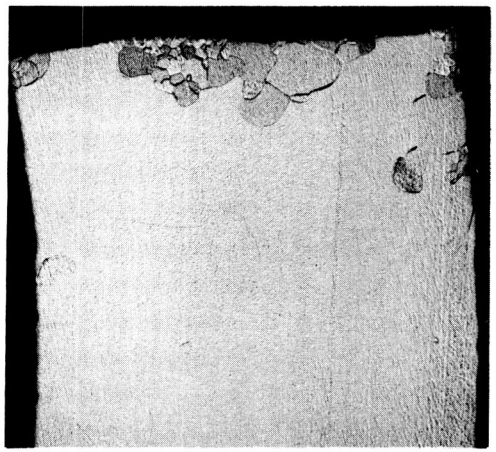
M-8255-1 (50×)

1800°C-100 hr



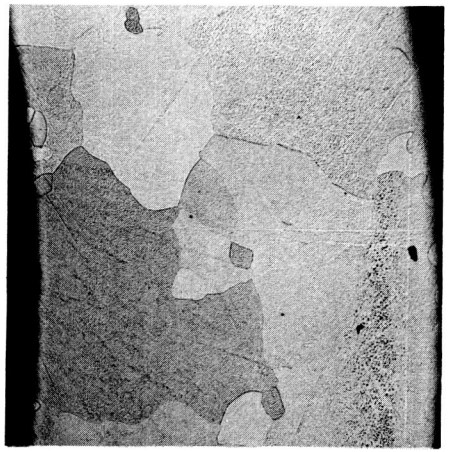
M-8485-1 (50×)

2000°C-15 hr



M-8543-1 (50×)

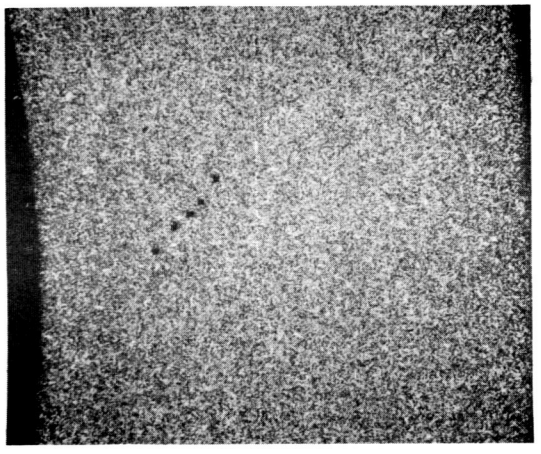
2200°C-15 hr



M-8590 (50×)

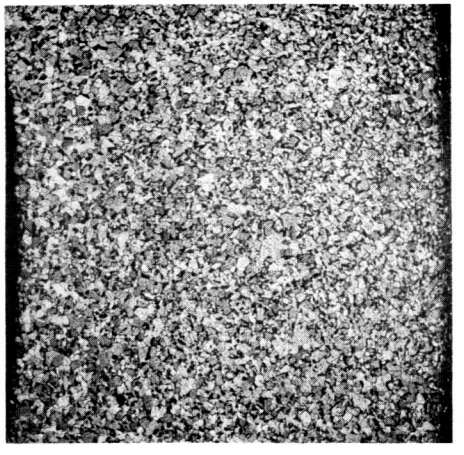
2500°C-1 hr

Fig. 13--Effect of thermal treatment on tungsten extruded at 1200°C (6 in. to 12 in. sections)--Tube No. 13



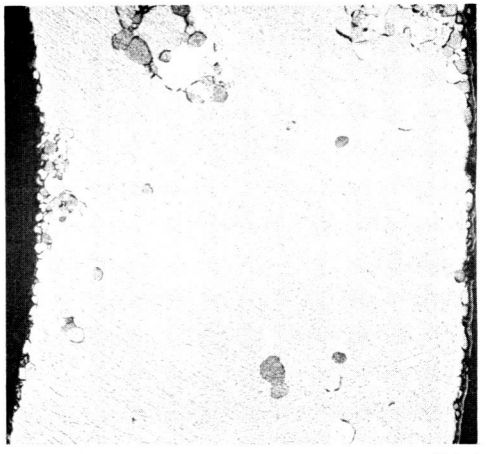
M-7964-1 (50×)

As-received



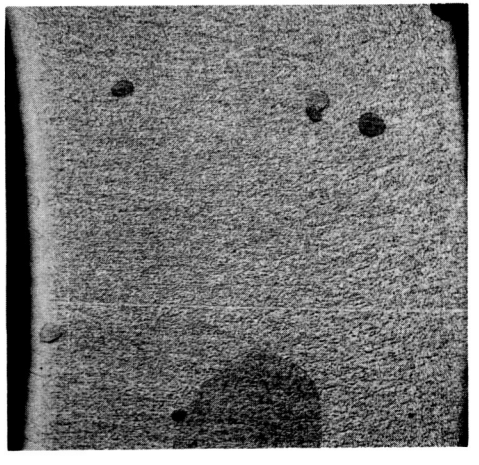
M-8256-1 (50×)

1800°C-100 hr



M-8486-1 (50×)

2000°C-15 hr



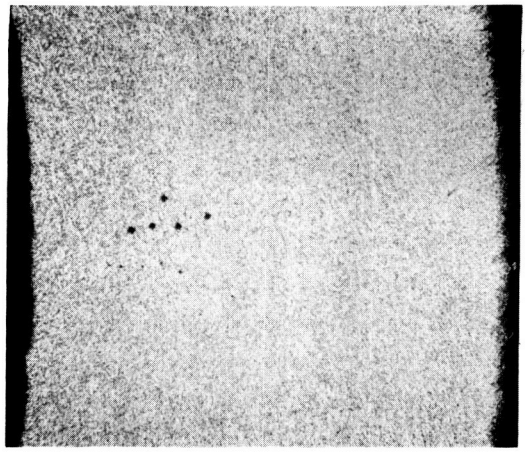
M-8544-1 (50×)

2200°C-15 hr

M-8591-1 (50×)

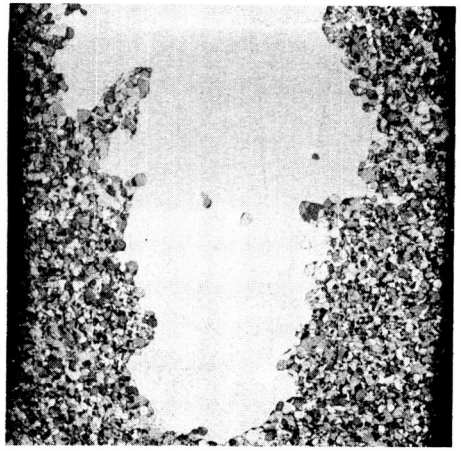
2500°C-1 hr

Fig. 14--Effect of thermal treatment on tungsten extruded at 1200°C (12 in. to 18 in. sections)--Tube No. 14



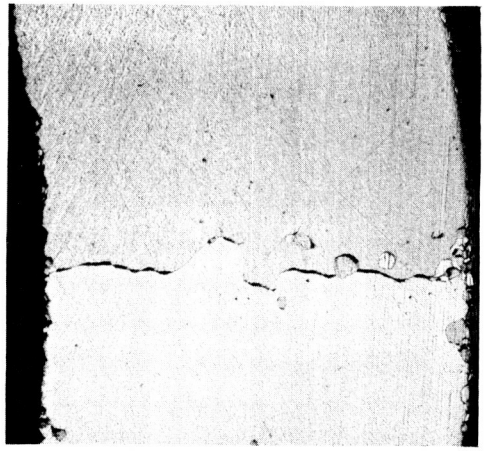
M-7965-1 (50×)

As-received



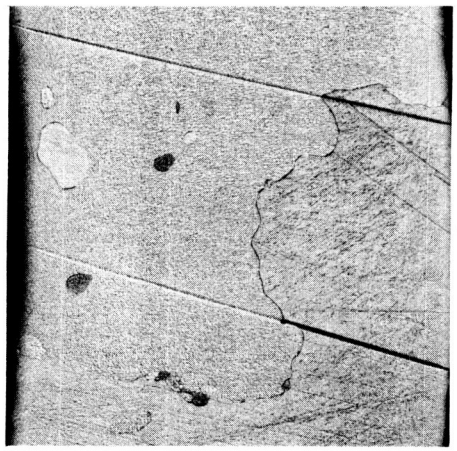
M-8257-1 (50×)

1800°C-100 hr



M-8487-1 (50×)

2000°C-15 hr



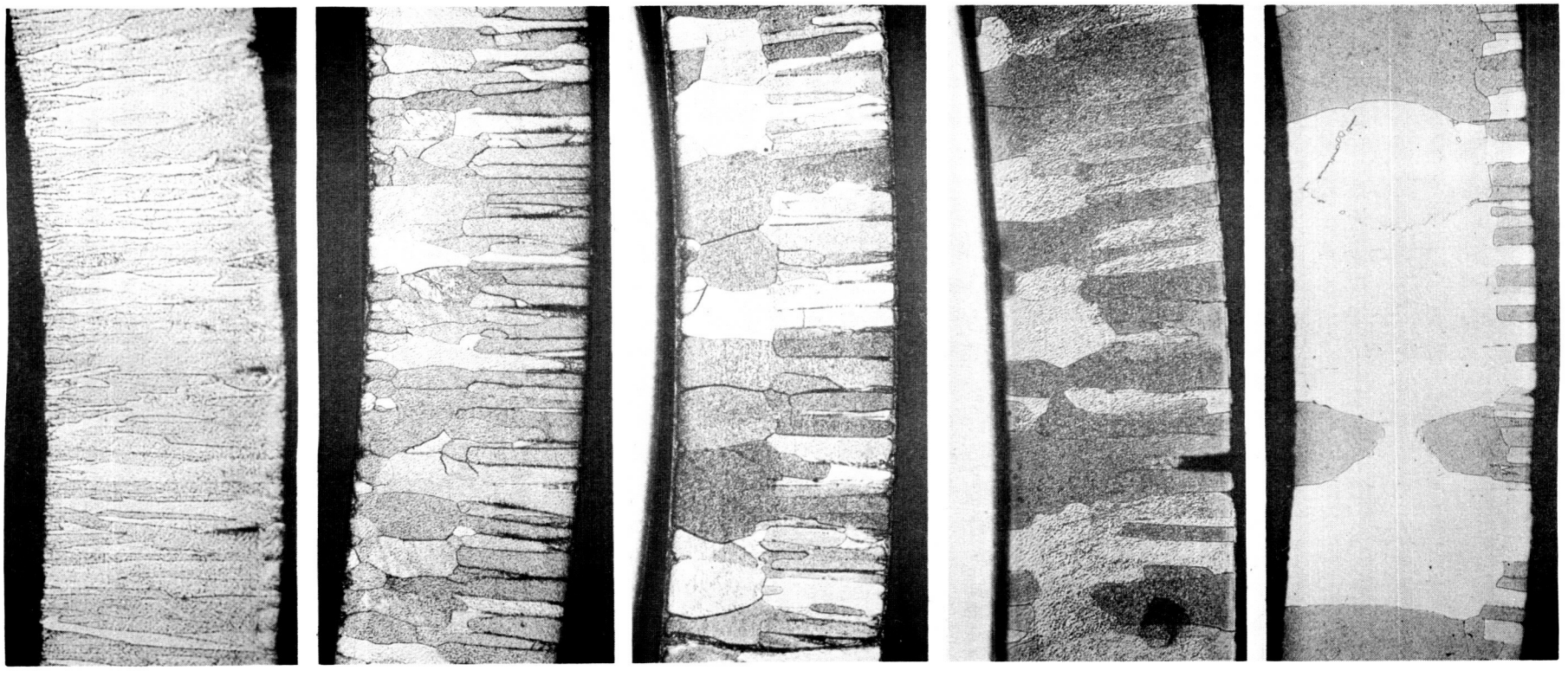
M-8545-1 (50×)

2200°C-15 hr

M-8592 1 (50×)

2500°C-1 hr

Fig. 15--Effect of thermal treatment on tungsten extruded at 1200°C (18 in. to 24 in. sections)--Tube No. 15



M-8177-1 (100×) As-received

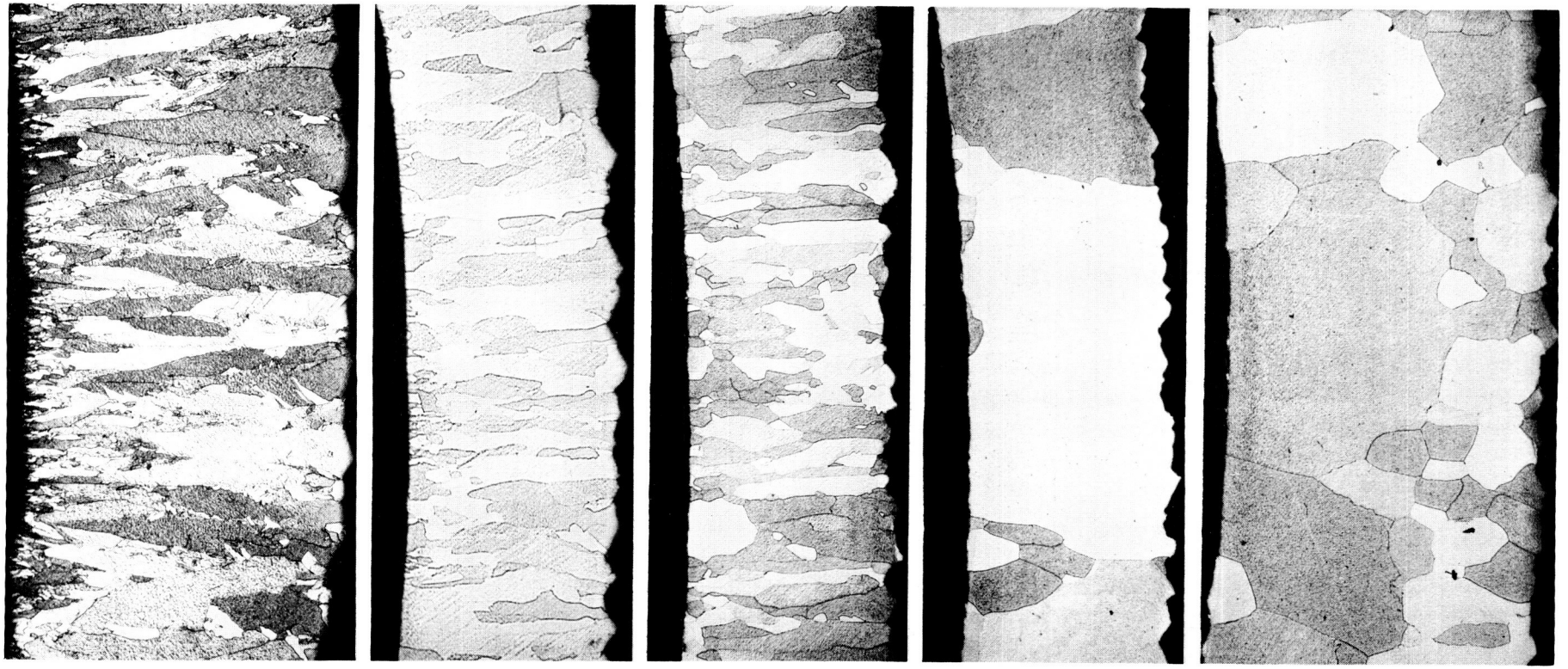
M-8258-1 (100×) 1800°C-100 hr

M-8488-1 (100×) 2000°C-15 hr

M-8546-1 (100×) 2200°C-15 hr

M-8830-1 (100×) 2500°C-1 hr

Fig. 16--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 16



M-8705-1 (100×) As-received | M-8891 (100×) 1800°C-100 hr | M-8827-1 (100×) 2000°C-15 hr | M-8828-1 (100×) 2200°C-15 hr | M-8829-1 (100×) 2500°C-1 hr

Fig. 17--Effect of thermal treatment on vapor-deposited tungsten--Tube No. 17

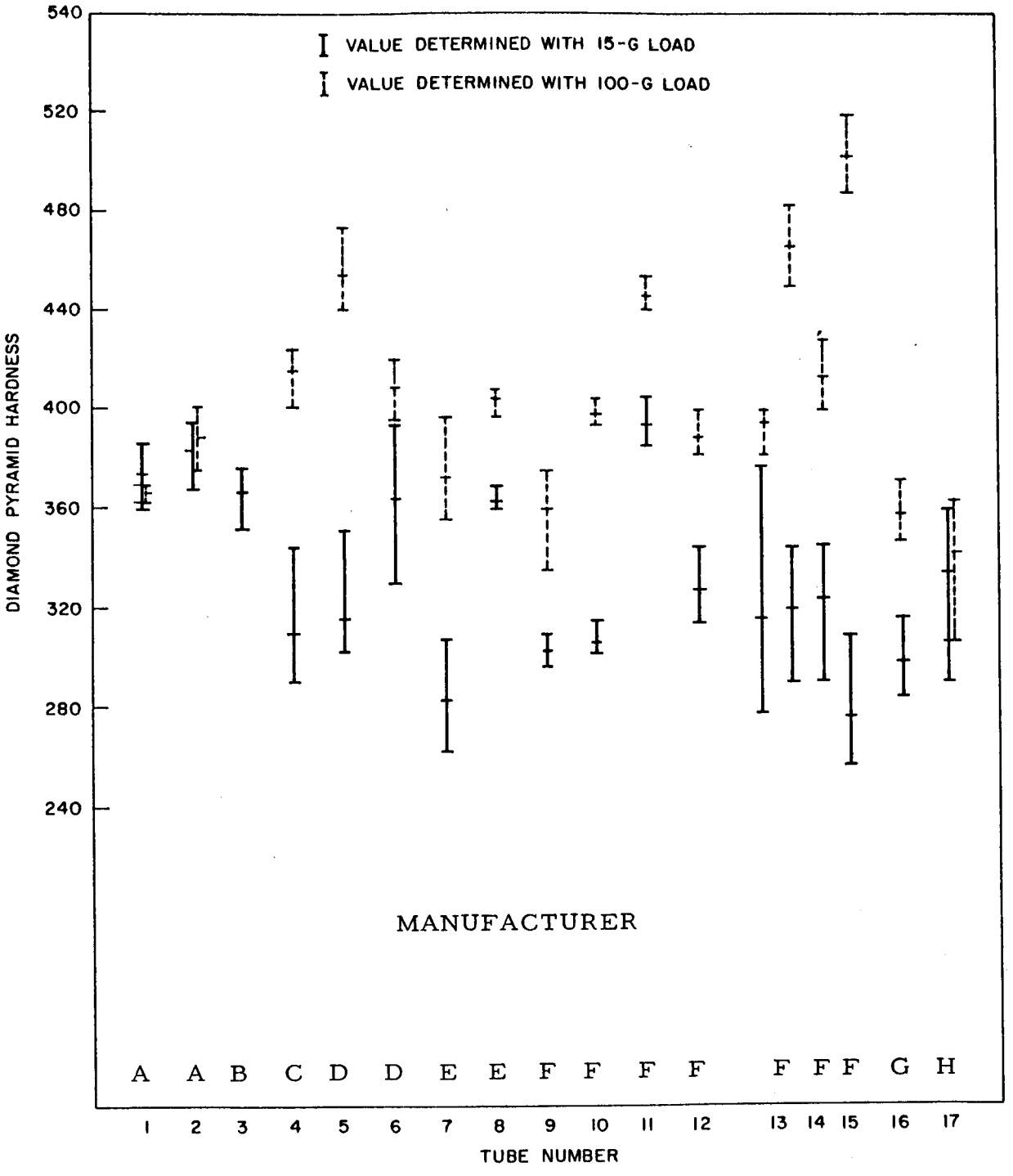


Fig. 18--Microhardness measurements on as-received tungsten tubing

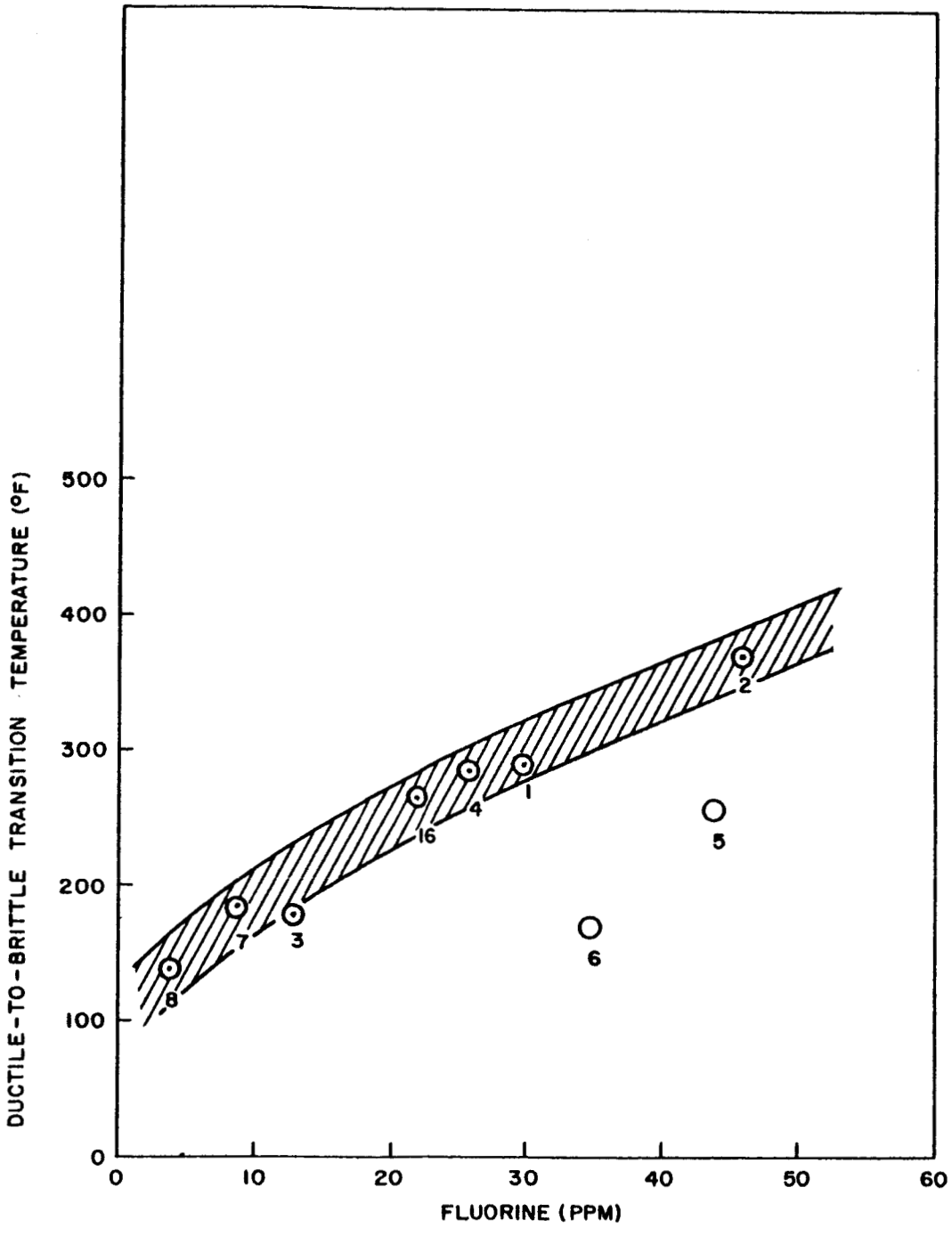


Fig. 19--Ductile-to-brittle transition temperature as a function of fluorine content (vapor-deposited tubing). Data point identification refers to tube numbers as shown in Table 1; data point 16 is plotted with respect to chlorine rather than fluorine content

Appendix A

LETTER OF INVITATION

The following is a copy of the letter that was sent to the fourteen organizations invited to submit samples of tungsten tubing for this evaluation.

"Gentlemen:

"For the past two years, as part of our program on thermionic energy conversion, we have been actively engaged in the development of vapor-deposited tungsten technology. As part of this program, we are initiating an evaluation of commercially available tungsten tubing and would like to invite you to submit a sample/or samples of tubing made by you for inclusion in these tests.

"The tests to be performed are:

1. Chemistry
   a) Gas content
   b) Carbon content
   c) Fluorine/or chlorine content
   d) Metallic impurities

2. Microstructure

3. Resistance to grain growth
   a) 10 hours at 1800°C
   b) 10 hours at 2000°C
   c) 10 hours at 2200°C
   d) 1 hour at 2500°C

4. Ductile-to-brittle transition temperature by ring bending tests.

"The results of all tests will be made available to you if you participate in this program, and will also be published as a report under our NASA sponsored contractual research programs.

"The size tubing we desire is nominally 3/8" O. D. × 0. 015" wall x 6" long. We have no objection to you selecting your 'best' material for these tests since we don't believe anybody knows what constitutes the best type of material with respect to all of the above tests. You may also submit two specimens which may be 'best' material with respect to different properties, i. e., one best for impurity content and one best for mechanical properties. Due to the number of specimens involved in the study not more than two specimens will be accepted from any one source.

"We would like to have a list of the process variables used in making your tubing (see enclosure 1), [enclosure 1 was a form for tabulation of the process variables] but being aware that proprietary information may be involved, this is NOT a requisite for submitting specimens for evaluation. However, we must at least know whether the fluoride, chloride, or some other process was used since this will influence the analytical chemical procedures employed.

"If necessary, we will pay your normal commercial price for the tubing you supply.

"For your information, a list of those being contacted is enclosed (see enclosure 2).

"If you are interested in submitting specimens for inclusion in this program, please reply before May 7, 1964, and include the cost of the tubing and the time after receipt of order required for delivery.

Very truly yours,

W. A. Shirley
Buyer"

Appendix B

ANALYTICAL PROCEDURES USED FOR IMPURITY CONTENT

The gaseous impurites N_2, O_2, and H_2 were determined by a vacuum-fusion analysis, the samples being dropped into a bath of iron at 1600°C. The carbon content was determined by a combustion-conductometric technique. The fluorine was determined spectrophotometrically using lanthanum-alizarin complexan, after a pyrohydrolytic separation. The metallic impurities were determined by means of an emission spectrograph. The chlorine was determined by neutron activation analysis.

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